

FORM PTO-1390 (Modified)  
(REV 11-98)

U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

ATTORNEY'S DOCKET NUMBER

**TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371**

**204403US0PCT**

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR

**09/806511**

INTERNATIONAL APPLICATION NO.

**PCT/FR99/02476**

INTERNATIONAL FILING DATE

**13 OCTOBER 1999**

PRIORITY DATE CLAIMED

**15 OCTOBER 1998**

TITLE OF INVENTION

**METHOD FOR PRODUCING A LAYER OF MATERIAL EMBEDDED IN ANOTHER MATERIAL**

APPLICANT(S) FOR DO/EO/US

**Bernard ASPAR, et al.**

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
  - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☒ has been transmitted by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
  - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
  - b. ☐ have been transmitted by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☐ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☒ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

**Items 13 to 20 below concern document(s) or information included:**

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☒ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ Certificate of Mailing by Express Mail
20. ☒ Other items or information:

**Request for Consideration of Documents Cited in International Search Report****Notice of Priority****Amended Sheets (pages 6, 7, 8, 14, 15, 16 and 17)****Drawings (1 sheet)****PCT/I B/304****PCT/IB/308**

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR <b>09/806511</b>		INTERNATIONAL APPLICATION NO. <b>PCT/FR99/02476</b>		ATTORNEY'S DOCKET NUMBER <b>204403US0PCT</b>	
--	--	--	--	---	--

21. The following fees are submitted:

BASIC NATIONAL FEE ( 37 CFR 1.492 (a) (1) - (5) ) :				CALCULATIONS PTO USE ONLY	
<input type="checkbox"/>	Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO .....	<b>\$1,000.00</b>			
<input checked="" type="checkbox"/>	International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO .....	<b>\$860.00</b>			
<input type="checkbox"/>	International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO .....	<b>\$710.00</b>			
<input type="checkbox"/>	International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) .....	<b>\$690.00</b>			
<input type="checkbox"/>	International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) .....	<b>\$100.00</b>			
<b>ENTER APPROPRIATE BASIC FEE AMOUNT =</b>					
			<b>\$860.00</b>		
Surcharge of <b>\$130.00</b> for furnishing the oath or declaration later than months from the earliest claimed priority date (37 CFR 1.492 (e)). <input type="checkbox"/> 20 <input type="checkbox"/> 30			<b>\$0.00</b>		
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	21 - 20 =	1	x \$18.00	<b>\$18.00</b>	
Independent claims	1 - 3 =	0	x \$80.00	<b>\$0.00</b>	
Multiple Dependent Claims (check if applicable). <input type="checkbox"/>				<b>\$0.00</b>	
<b>TOTAL OF ABOVE CALCULATIONS =</b>				<b>\$878.00</b>	
Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) (check if applicable). <input type="checkbox"/>				<b>\$0.00</b>	
<b>SUBTOTAL =</b>				<b>878</b>	
Processing fee of <b>\$130.00</b> for furnishing the English translation later than months from the earliest claimed priority date (37 CFR 1.492 (f)). <input type="checkbox"/> 20 <input type="checkbox"/> 30			+	<b>\$0.00</b>	
<b>TOTAL NATIONAL FEE =</b>				<b>\$878.00</b>	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) (check if applicable). <input type="checkbox"/>				<b>\$0.00</b>	
<b>TOTAL FEES ENCLOSED =</b>				<b>\$878.00</b>	
				Amount to be: refunded	\$
				charged	\$


☒ A check in the amount of **\$878.00** to cover the above fees is enclosed.

☐ Please charge my Deposit Account No. \_\_\_\_\_ in the amount of \_\_\_\_\_ to cover the above fees.  
A duplicate copy of this sheet is enclosed.

☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **15-0030** A duplicate copy of this sheet is enclosed.

**NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.**

SEND ALL CORRESPONDENCE TO:



**22850**

(703) 413-3000

Surinder Sachar  
Registration No. 34,423

*Surinder Sachar*

SIGNATURE

**Norman F. Oblon**

NAME

**24,618**

REGISTRATION NUMBER

*April 16 2001*

DATE

204403US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF:

:

BERNARD ASPAR ET AL.

:

SERIAL NO: NEW US PCT APPLN.  
(Based on PCT/FR99/02476)

: ATTN: APPLICATION BRANCH

FILED: HEREWITH

:

FOR: METHOD FOR PRODUCING A  
LAYER OF MATERIAL EMBEDDED  
IN ANOTHER MATERIALPRELIMINARY AMENDMENTASSISTANT COMMISSIONER FOR PATENTS  
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows.

IN THE CLAIMS

Please cancel Claims 1-21 and add the following claims:

- 22. (New) A method for producing a layer of a first material embedded in a substrate comprising at least one second material, comprising the following stages:
- formation in said substrate, at the level of the desired embedded layer, and by a method excluding the formation of a porous layer, of a layer of microcavities intended to serve as centers of nucleation and volume accommodation to produce said first material in said second material,

- formation of precipitate embryos from the nucleation centers formed, the precipitate embryos corresponding to the first material,

- growth of the precipitates from the embryos through species concentration corresponding to the first material and carried to the microcavity layer.

23. (New) The method according to Claim 22, in which the layer of microcavities is formed by introducing gaseous species into the second material.

24. (New) The method according to Claim 23, in which the gaseous species used to form the layer of microcavities are chosen from among hydrogen, helium and fluorine.

25. (New) The method according to Claim 22, in which the layer of microcavities is formed by an inclusion of gas provoked during formation of the substrate.

26. (New) The method according to Claim 22, in which the layer of microcavities is formed from the interface constituted by the solidarization of a first substrate element and a second substrate element, providing said substrate.

27. (New) The method according to Claim 26, in which the layer of microcavities results from the presence of particles at said interface.

28. (New) The method according to Claim 26, in which the layer of microcavities results from the surface roughness of at least one element among the first substrate element and the second substrate element.

29. (New) The method according to Claim 26, in which the layer of microcavities results from the presence of micro-recesses at the surface of at least one element among the first substrate element and the second substrate element.

30. (New) The method according to Claim 26, in which the layer of microcavities results from stresses induced at said interface.

31. (New) The method according to Claim 22, in which the precipitate embryos are formed from species present in the second material.

32. (New) The method according to Claim 22, in which the precipitate embryos are formed from species introduced into the second material.

33. (New) The method according to Claim 32, in which said introduction is carried out by thermally activated diffusion.

34. (New) The method according to Claim 33, in which, the formation of microcavities implementing a thermal treatment, the precipitate embryos are formed simultaneously with the microcavities.

35. (New) The method according to Claim 22, in which the growth of the precipitates is produced by concentration of species introduced into the substrate.

36. (New) The method according to Claim 35, in which the growth of the precipitates is produced by concentration of species introduced into the substrate by thermally activated diffusion.

37. (New) The method according to Claim 35, in which the growth of the precipitates is produced by concentration of species introduced under pressure into the substrate.

38. (New) The method according to Claim 35, in which the growth of the precipitates is produced by concentration of species introduced into the substrate by means of a plasma.

39. (New) The method according to Claim 22, in which the growth of the precipitates is produced by concentration of species present in the substrate, under the effect of a thermal treatment.

40. (New) The method according to Claim 22, in which the formation of precipitate embryos and the growth of precipitates being two operations requiring a thermal treatment, these operations are carried out simultaneously.

41. (New) The method according to Claim 22 in which the layer of microcavities is formed in a semiconductor substrate.

42. (New) The method according to Claim 41, in which the substrate is in silicon and the embedded layer is a layer of silicon oxide.--

#### REMARKS

Claims 22-42 are active in the present application. Support for these claims is found in Claims 1-21 and the specification as filed herewith. No new matter is believed to have been added by these amendments. An action on the merits and allowance of the claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,  
MAIER & NEUSTADT, P.C.



Norman F. Oblon  
Attorney of Record  
Registration No. 24,618

Daniel J. Pereira, Ph.D.  
Registration No. 45,518



**22850**

(703) 413-3000

NFO/DJP/smi

I:\user\DJPER\204403US-pr.wpd

METHOD FOR PRODUCING A LAYER OF MATERIAL EMBEDDED IN  
ANOTHER MATERIAL

Technical field

The present invention relates to a method for producing a layer of material embedded in another material. It is applicable in particular to the field of semiconductors and especially for producing  
5 substrates of the Silicon on Insulator type.

At present, substrates of the Silicon on Insulator or SOI type are of very great interest for micro-electronic applications in the domain of low consumption. There are several methods for obtaining  
10 SOI substrates. Those used most these days are the SIMOX process (Separation by Implanted Oxygen) and methods based on bonding by molecular adhesion (wafer bonding). In order to obtain thin films of silicon on silica, these methods, using wafer bonding, are  
15 combined with thinning methods. As a thinning process, one can quote that revealed in document FR-A-2 681 472 where the cleavage of a substrate is obtained by coalescence, provoked by a thermal treatment of microcavities generated by ion implantation. One can  
20 also cite processes using epitaxied barrier layers and selective etching.

State of prior art

It is known that implantation by bombardment of an  
25 inert gas or hydrogen in a semiconductor material (see FR-A-2 681 472), or in a solid material, crystalline or not (see FR-A-2 748 850), is capable of creating

microbubbles (called platelets or nanoblister) at a depth close to the average depth of ion penetration. The morphology (dimension, shape...) of these defects can change during thermal treatments, in particular the size of these cavities can increase. Depending on the nature of the material and above all its mechanical properties, these cavities, present at the average depth of penetration of gas species can, depending on the thermal treatment conditions, induce surface deformations or blisters. The most important parameters to monitor to obtain these blisters are the gas dose introduced during implantation, the depth to which the gas species are implanted and the total thermal budget supplied to the material. In certain cases, the conditions of implantation are such that, after annealing, microcavities or microbubbles are present at the level of the average depth of ion implantation but their size and pressure inside these cavities are not sufficient to induce surface deformations. Then there is a continuous layer of defects embedded without any degradation of the surface. As an example, implantation of hydrogen in a silicon plate according to a dose of  $3 \cdot 10^{16} \text{H}^+/\text{cm}^3$  and an energy of 25 keV creates a continuous embedded layer of microcavities of about 150 nm thickness at an average depth of about 300 nm. These microcavities have an elongated shape: their size is of the order of 6 nm in length and two atomic planes in thickness. If annealing is carried out at 600°C for 30 minutes on this plate, the microcavities grow and their size changes from 6 nm to more than 50 nm in length and from a few atomic planes to 4-6 nm in



thickness. On the other hand, no surface disturbance is observed.

The presence of microcavities can also be seen in the case of implantation by helium bombardment, at the level of the average implantation depth ( $R_p$ ) in silicon. In this case, the cavities have a stable form which does not change during annealing. Reference can be made to the article "Radiation damage and implanted He atom interaction during void formation in silicon" by V. RAINERI and M. SAGGIO, Appl. Phys. Lett. 71 (12), 22 September 1997.

Furthermore, it is known that defects present in materials are the preferential nucleation centres for the formation of a heterogeneous phase. As an example, concerning the formation of oxide precipitates, three types of nucleation are listed in the bibliography: in the homogeneous phase, in the homogeneous phase under stresses, in heterogeneous phases (see for example the article entitled "Oxygen Precipitation in Silicon" by A. BORHESI et al., J. Appl. Phys. 77(9), 1995, pages 4169-4244). This oxygen which precipitates is contained in the initial material. It comes, for example, from the formation stage of the material.

By nucleation is meant the formation of aggregates of several oxygen atoms in silicon to form nucleation centres called "nuclei" or "precipitate embryos". More simply, nucleation can appear in crystalline sites corresponding to knots of the network where several interstitial oxygen atoms are close to each other (homogeneous nucleation) or on network defects (heterogeneous nucleation). It is known that these

network defects can be point defects induced by the presence of an element exterior to the matrix (for example carbon in the silicon) or complexes such as, for example, oxygen-carbon complexes (see the article mentioned above by A. BORHESI et al.). For example, the point defects intrinsic to the material such as clusters of vacancies formed during silicon growth can also be nucleation centres for obtaining "nuclei". Furthermore, as an example of defects induced by the presence of an external element, the case can be cited of carbon introduced into the substrate to create a continuous embedded layer rich in carbon which will act as a nucleation zone. The introduction of carbon can be obtained by implantation of carbon by bombardment.

After the formation phase of these nucleation centres, to obtain a precipitate of larger size, it is necessary to have a precipitation phase. Precipitation in a material is a phenomenon of aggregation of atoms to form small particles or precipitates.

The critical radius  $r_c$  defining the minimum size of precipitates capable of existing is given, for a concentration of interstitial oxygen in the material, by the equation  $r_c = (2\sigma/\Delta H) (T_s/T_s - T)$  where

$\sigma$  is the surface energy,  
 $\Delta H$  is the formation enthalpy,  
 $T$  is the temperature expressed in Kelvins  
 $T_s$  is the equilibrium temperature corresponding to the given quantity of oxygen,

(see the article "Oxygen Precipitation Czochralski Silicon" by R. CRAVEN, Elec. Chem. Soc., Proceedings of

the 4th Int. Symp. on Silicon Materials, Science and Technology Vol. 81-5, 1981).

Starting from this equation, it can be clearly seen that the rise in temperature brings about growth  
5 of the precipitates.

To resume, defects create nucleation centres, which will serve to form precipitates which will then become bigger.

On the other hand, studies have demonstrated the  
10 possibility of reducing the number of discontinuities of the oxide layer embedded in the case of SOI substrates obtained by the "low dose" SIMOX process with the help of oxidation at high temperature (over 1350°C) of the silicon film (see patent US 5 589 407  
15 and the article entitled "An Analysis of Buried-Oxide Growth in Low-dose SIMOX Wafers by High-Temperature Thermal Oxidation" vt S. MASUI et al., Proceedings 1995 IEEE International SOI Conference, October 1995). This process, named ITOX (Internal Oxidation), makes it  
20 possible to oxidise the oxide layer embedded by means of oxygen diffusion from the surface to the embedded oxide layer. Other authors demonstrate that the same phenomenon occurs at lower temperatures, of the order of 1200°C (see article "Internal Oxidation of Low Dose  
25 Separation by Implanted Oxygen Wafers in Different Oxygen/Nitrogen Mixtures" by P. ERICSSON and S. BENGTSSON, accepted for publication in Appl. Phys. Lett.).

The latter results indicate that the introduction  
30 of oxygen into the material depends firstly on the time applied at high temperature and not on the quantity of

English translation of the amended sheets of  
International Preliminary Examination Report

oxygen in the annealing atmosphere. Thus it seems that the introduction of oxygen may be limited by the solubility limit of oxygen in silicon. Thus, the higher the temperature, the faster the oxidation effect for the embedded oxide layer. An example of this phenomenon indicates that at 1200°C, if 5% oxygen is introduced into nitrogen, 8 hours of annealing allow the embedded oxide layer to grow in thickness from 860 Angströms to 1330 Angströms. This "internal" oxidation is of interest because it reduces the density of discontinuities of the embedded oxide.

From the abstract of the document JP-A-56 110 247 a method is known for the formation of an insulating region inside a semiconductor substrate of type N. The substrate is first irradiated by light elements making it possible to create an embedded region of type P in the substrate. This region of type P is then made porous by an anodic treatment. Thermal treatment under oxygen makes the porous region insulating.

The document "Silicon on Insulator Structures Obtained by Epitaxial Growth of Silicon over Porous Silicon" by C. OULES et al., in R. Electrochem. Soc. Vol. 139, No. 12, December 1992, reveals the production of a SOI structure comprising a silicon substrate showing a porous layer formed by anodisation and supporting an epitaxial layer of silicon.

Description of the invention

The invention proposes a new process for making an embedded layer of material in a substrate of another material. An original aspect of the invention consists of creating microcavities embedded in the substrate and not crystalline defects for creating traps. In particular, when the substrate is in silicon, this makes it possible to obtain a superficial layer of silicon of much higher quality and does not require the substrate to be submitted to a temperature of the order of 1300°C in order to mend the defects created by oxygen implantation, for example. of 1300°C in order to mend the defects created by oxygen implantation, for example.

The present invention also offers the advantage of being able to implement an implantation with a low dose of a light element (for example hydrogen) which does not induce a crystalline defect between the surface implanted and the microcavity creation zone, contrary to what happens when ions are implanted, such as oxygen, silicon or argon. This process is simple to

English translation of the amended sheets of  
International Preliminary Examination Report

implement, since the implantation can take place  
at ambient temperature.

The aim of the invention is thus a process for  
producing a layer of a first material embedded in a  
5 substrate comprising at least one second material,  
characterised in that it comprises the following  
stages:

- formation of said substrate, at the level of the  
desired embedded layer, of a layer of microcavities  
10 (called "platelets" or microbubbles) intended to serve  
as centres of nucleation and volume accommodation to  
produce said first material in said second material,

- formation of precipitate embryos from the  
nucleation centres formed, the precipitate embryos  
15 corresponding to the first material,

- growth of the precipitates from the embryos  
through species concentration corresponding to the  
first material and carried to the microcavity layer.

The microcavity layer can be formed advantageously  
20 by introducing into the second material gaseous  
species, which are advantageously chosen from among  
hydrogen, helium and fluorine. Furthermore one can form  
the layer of microcavities by a gas inclusion provoked  
during the production of the substrate. It can also be  
25 formed from the interface constituted by solidarisation  
of a first substrate element and a second substrate  
element, supplying said substrate. The microcavity  
layer can then result from the presence of particles at  
said interface, from the

30

English translation of the amended sheets of  
International Preliminary Examination Report

surface roughness of at least one element from the first substrate element and the second substrate element, from the presence of micro-recesses at the surface of at least one element from the first substrate element and the second substrate element or stresses induced at said interface.

The precipitate embryos can be formed from species present in the second material. They can also be formed from species introduced in the second material. This introduction can be carried out by diffusion activated thermally. In this case, if the formation of microcavities implements a thermal treatment, the precipitate embryos can be formed simultaneously with the microcavities.

The growth of precipitates can be carried out by concentration of species introduced into the substrate. This introduction can be carried out by thermally activated diffusion, under pressure or by means of a plasma.

The growth of precipitates can be carried out by concentration of species present in the substrate, under the effect of a thermal treatment.

If the formation of precipitate embryos and the growth of these precipitates are two operations requiring thermal treatment, these operations can be carried out simultaneously.

The invention is related in particular to the production of a semiconductor substrate provided with an embedded layer. It applies in particular to the production of a silicon substrate provided with an embedded layer of silicon oxide.

The embedded layer can be continuous or not depending on the applications foreseen. In this case, one can play on the density of the precipitates, and the utilisation of a mask protecting certain zones of the material submitted to the process according to the invention.

#### Short description of the drawing

The invention will be better understood and other advantages and details will become apparent by reading the following description, given as a non-limiting example, accompanied by the attached drawing which represents, in a transversal cross-section, a substrate in which there is an embedded layer of a material different from the material constituting the substrate.

#### Detailed description of the embodiments of the invention

As an example, a description is given below of a process for producing a SOI substrate according to the present invention, starting from a solid silicon substrate.

The first stage consists of forming a layer of microcavities in the substrate reference 1 on the attached figure.

A technique which is simple to operate consists of forming this layer of microcavities by hydrogen bombardment at doses (for example  $3 \cdot 10^{16} \text{H}^+/\text{cm}^2$ ) which makes it possible to obtain at the average penetration depth of  $R_p$  particles, and after annealing at  $600^\circ\text{C}$  for 30 minutes, elongated microcavities with the length of

several tens of nanometres. If the implantation energy is of the order of 50 keV, the average depth  $R_p$  of the layer of microcavities 2 is about 500 nm from the face 3 of the substrate through which the implantation is carried out. The width of the layer of microcavities 2 is then of the order of 150 nm.

The dose couple of hydrogen implanted/annealing formation of microcavities is highly dependent on the implantation parameters and in particular on the implantation temperature.

By using the term hydrogen, one means the gaseous species constituted under their atomic form, under their molecular form, under their ion form, under their isotopic form (deuterium) or yet again under their isotopic and ion form.

The SOI substrate required to have an embedded layer of silicon oxide, the phase for creation of oxide precipitate embryos can be produced from the oxygen present in the silicon of the substrate, by means of thermal treatment at a temperature comprised between 750°C and 800°C. Given the fact that one wishes to obtain an embedded oxide layer, it is preferable that the annealing atmosphere contains oxygen. In this case, a small thickness of oxide forms at the surface of the substrate. This superficial oxide layer can be eliminated at the end of the process according to the invention.

Once the precipitate embryos are formed, they are made to grow by means of oxidation annealing. In this case two parameters have to be taken into account: the



quantity of oxygen introduced into the material and the critical radius of the oxide precipitates.

The quantity of oxygen introduced into the material is controlled by the solubility limit of oxygen in silicon. The higher the temperature the higher the solubility limit.

The critical radius of oxide precipitates is increased proportionally when the temperature is raised.

Consequently, if one wishes to grow precipitates of small dimensions and not dissolve them, it is necessary to carry out the thermal treatment at a temperature lower than the degradation temperature of the precipitate. Therefore, a satisfactory compromise has to be found. One solution is to employ long annealing times with slow rises in temperature. As an example, one can proceed as follows:

- a first stage at 750°C for 2 hours,
- a second stage at 800°C for 3 hours,
- a third stage at 900°C for 2 hours,
- a fourth stage at 1000°C for 2 hours,
- a fifth stage at 1100°C for 2 hours,
- a sixth stage at 1200°C for 8 hours, before reducing the temperature.

Another important point to be monitored is the annealing atmosphere. If it is necessary to remain at 1200°C for a long time to introduce oxygen into the silicon matrix, the substrate will also be oxidised substantially on its surface. In order to keep a maximum of surface silicon, it is necessary to use an atmosphere low in oxygen, for example 5% oxygen diluted

in nitrogen. In this case, for a stage of 8 hours at 1200°C, the superficial oxide thickness is of the order of 120 nm and the embedded oxide layer formed is then about 50 nm. Thus one obtains an embedded layer 2 of oxide of 50 nm thickness under a thin film 4 of silicon of about 350 nm.

The thickness of the oxide layer 2 and the thin film 4 are thus conditioned by the implantation energy and the annealing conditions (atmosphere, length of time, temperature).

A variant of the procedure can consist of implanting helium with a dose of  $2 \cdot 10^{16} \text{He}^+/\text{cm}^2$  under an implantation energy of 100 keV and of carrying out annealing at 900°C for 55 minutes to obtain a layer of microcavities at an average depth of about 600 nm for the implanted surface. The silicon substrate can then be oxidised in conditions (temperature and time length steps) identical to those described above, but beginning directly at 900°C. Thus one obtains, by the same procedure as before, an embedded oxide layer of 50 nm thickness under a silicon film of about 450 nm thickness.

Thus the invention makes it possible to obtain, from a solid substrate 1 in silicon, a SOI substrate constituted of a plate 5 of silicon covered with an insulating layer 2 itself covered with a thin film 4 of silicon.

Among the advantages of the process according to the invention, one can mention the fact that it makes it possible to use a single slice of the same material to provide a SOI substrate. It gives very good

2

100

English translation of the amended sheets of  
International Preliminary Examination Report

CLAIMS

1. Method for producing a layer (2) of a first material embedded in a substrate (1) comprising at least one second material, characterised in that it comprises the following stages:

- 5           - formation in said substrate (1), at the level of the desired embedded layer and by a method excluding formation of a porous layer, of a layer of microcavities intended to serve as centres of nucleation and volume accommodation to produce said
- 10 first material in said second material,
- formation of precipitate embryos from the nucleation centres formed, the precipitate embryos corresponding to the first material,
- growth of the precipitates from the embryos
- 15 through species concentration corresponding to the first material and carried to the layer of microcavities.

2. Method according to Claim 1, characterised in

20 that the layer of microcavities is formed by introducing gaseous species into the second material.

3. Method according to Claim 2, characterised in that the gaseous species used to form the layer of

25 microcavities are chosen from among hydrogen, helium and fluorine.

English translation of the amended sheets of  
International Preliminary Examination Report

4. Method according to Claim 1, characterised in that the layer of microcavities is formed by a gas inclusion provoked during production of the substrate.

5 5. Method according to Claim 1, characterised in that the layer of microcavities is formed from the interface constituted by the solidarisation of a first substrate element and a second substrate element, providing said substrate.

10

6. Method according to Claim 5, characterised in that the layer of microcavities results from the presence of particles at said interface.

15

7. Method according to Claim 5, characterised in that the layer of microcavities results from the surface roughness of at least one element among the first substrate element and the second substrate element.

20

8. Method according to Claim 5, characterised in that the layer of microcavities results from the presence of micro-recesses at the surface of at least one element among the first substrate element and the  
25 second substrate element.

30

9. Method according to Claim 5, characterised in that the layer of microcavities results from stresses induced at said interface.

English translation of the amended sheets of  
International Preliminary Examination Report

10. Method according to Claim 1, characterised in that the precipitate embryos are formed from species present in the second material.

5 11. Method according to Claim 1, characterised in that the precipitate embryos are formed from species introduced into the second material.

10 12. Method according to Claim 11, characterised in that said introduction is carried out by thermally activated diffusion.

15 13. Method according to Claim 12, characterised in that, the formation of microcavities implementing a thermal treatment, the precipitate embryos are formed simultaneously with the microcavities.

20 14. Method according to Claim 1, characterised in that the growth of the precipitates is produced by concentration of species introduced into the substrate (1).

25 15. Method according to Claim 14, characterised in that the growth of the precipitates is produced by concentration of species introduced into the substrate by thermally activated diffusion.

30 16. Method according to Claim 14, characterised in that the growth of the precipitates is produced by concentration of species introduced under pressure into the substrate.

English translation of the amended sheets of  
International Preliminary Examination Report

17. Method according to Claim 14, characterised in  
that the growth of the precipitates is produced by  
concentration of species introduced into the substrate  
5 by means of a plasma.

18. Method according to Claim 1, characterised in  
that the growth of the precipitates is produced by  
concentration of species present in the substrate,  
10 under the effect of a thermal treatment.

19. Method according to Claim 1, characterised in  
that the formation of precipitate embryos and the  
growth of precipitates being two operations requiring a  
15 thermal treatment, these operations are carried out  
simultaneously.

20. Method according to any one of Claims 1 to 19,  
characterised in that the layer of microcavities is  
20 formed in a semiconductor substrate.

21. Method according to Claim 20, characterised in  
that the substrate (1) is in silicon and that the  
embedded layer (2) is a layer of silicon oxide.

ABSTRACT OF THE DISCLOSUREMETHOD FOR PRODUCING A LAYER OF MATERIAL EMBEDDED IN  
ANOTHER MATERIAL

The aim of the invention is a method for producing a layer (2) of a first material embedded in a substrate (1) comprising at least one second material. The method comprises the following stages:

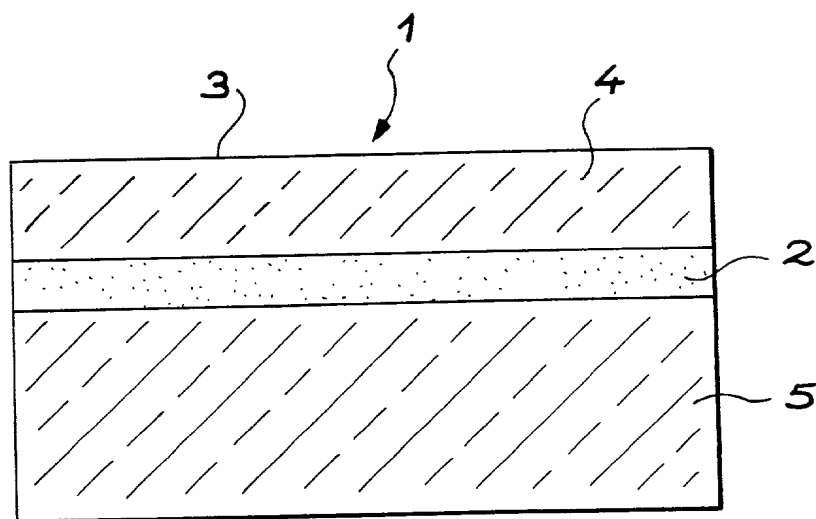
- 5       - formation in the substrate (1), at the level of the desired embedded layer, of a layer of microcavities intended to serve as centres of nucleation to produce said first material in said second material,
- formation of precipitate embryos from the  
10 nucleation centres formed, the precipitate embryos corresponding to the first material,
- growth of the precipitates from the embryos through species concentration corresponding to the first material and carried to the microcavity layer.

15

Single figure



1 / 1



# Declaration, Power Of Attorney and Petition

WE (I) the undersigned inventor(s), hereby declare(s) that :

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled  
METHOD FOR PRODUCING A LAYER OF MATERIAL EMBEDDED IN ANOTHER MATERIAL

the specification of which

- ☐ is attached hereto.
- ☐ was filed on  
as Application Serial No.  
and amended on
- ☒ was filed as PCT international application  
Number PCT/FR99/02476  
on October 13, 1999  
and was amended under PCT Article 19  
on October 23, 2000

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119 (a)-(d) or § 365 (b) of any foreign application(s) for patent or inventor's certificate, or § 365 (a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application (s)

Application No.	Country	Day/month/Year	Priority Claimed	
98 12950	FRANCE	15 OCTOBER 1998	<input checked="" type="checkbox"/> YES	<input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES	<input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES	<input type="checkbox"/> NO
_____	_____	_____	<input type="checkbox"/> YES	<input type="checkbox"/> NO

We (I) hereby claim the benefit under Title 35, United States Code, § 119 (e) of any United States provisional application(s) listed below.

\_\_\_\_\_  
(Application Number)

\_\_\_\_\_  
(Filing Date)

\_\_\_\_\_  
(Application Number)

\_\_\_\_\_  
(Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of prior application and the national or PCT International filing date of this application.

Application Serial No.

Filing Date

Status (pending, patented,  
abandoned)

And we (I) hereby appoint : Norman F. Oblon, Registration Number 24,618; Marvin J. Spivak, Registration Number 24,913; C. Irvin McClelland, Registration Number 21,214; Gregory J. Maier, Registration Number 25,599; Arthur I. Neustadt, Registration Number 24,854; Richard D. Kelly, Registration Number 27,757; James D. Hamilton, Registration Number 28,421; Eckhard H. Kuesters, Registration Number 28,870; Robert T. Pous, Registration Number 29,099; Charles L. Gholz, Registration Number 26,395; Vincent J. Sunderdick, Registration Number 29,004; William E. Beaumont, Registration Number 30,996; Steven B. Kelber, Registration Number 30,073; Robert F. Gnuse, Registration Number 27,295; Jean-Paul Lavalleye, Registration Number 31,451; William B. Walker, Registration Number 22,498; Timothy R. Schwartz, Registration Number 32,171; Stephen G. Baxter, Registration Number 32,884; Martin M., Zoltick, Registration Number 35,745; Robert W. Hahl, Registration Number 33,893; and Richard L. Treanor, Registration Number 36,379; our (my) attorneys, with full powers of substitution and revocation, to prosecute this application and to transact all business in the Patent Office connected therewith; and we (I) hereby request that all correspondence regarding this application be sent to the firm of OBLON, SPIVAK, McCLELLAND, MAIER & NEUSTADT, P.C., whose post Office Address is : Fourth Floor, 1755 Jefferson Davis Highway, Arlington, Virginia 22202.

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true ; and future that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardise the validity of the application or any patent issuing thereon.

1-00 ASPAR Bernard.

NAME OF FIRST SOLE INVENTOR

Signature of Inventor

February 27, 2001  
Date

Residence : 110 Lot le Hambeau  
des Ayes  
38140 RIVES - France

Citizen of : Française

Post Office Address : The same as residence

~~Signature of Inventor~~

Residence : PRESIDENT N°9

38 113 VEUREY ~~FR~~  
FRANCE

Citizen of: FRANCE

MORICEAU Hubert

Signature of Inventor

Residence : 26 Rue du fournet

38120 St Egreve - FRANCE

Citizen of: Française

Post Office Address : The same as residence

NAME OF FOURTH INVENTOR

Signature of Inventor

Date \_\_\_\_\_

NAME OF FIFTH INVENTOR

Signature of Inventor

Date \_\_\_\_\_

Residence :

Citizen of: \_\_\_\_\_

**Post Office Address :** The same as residence

Residence : \_\_\_\_\_

Citizen of :

Post Office Address : The same as residence